Users' Executive Committee and NSLS Town Meetings

Mark Chance
Users' Executive Committee Chair, Albert Einstein College of Medicine

The UEC, or sers Executive Committee, represents the interests of the users to NSLS and BNL Administrations as well as to the community outside BNL. Those interests include continued access to reliable X-ray and UV beams and to a working environment supportive of their research. Almost 2500 users carried out experiments at the NSLS during 2000. "Users" are defined as researchers who actually showed up at the User Administration Office and were given access to the experimental floor to perform experiments on a beamline. With over 95-98% reliability on both storage rings, most users are satisfied that the NSLS can provide what they need to fulfil their research obligations.

However, the user community is changing. Although the number of users classifying themselves as material scientists has remained steady the last several years, it has not grown significantly for some time. the numbers of users classifying themselves as biology or environmental and geological sciences continues to grow significantly. The numbers of these users are 10 times what they were 10 years ago and the number of biology users is now over 900, is the largest single group for the second year in a row, and continues to rise. To a significant degree, the materials science and physics users are savvy about beamlines and knowledgeable about some or all of the physics that goes into an accelerator facility and its beamlines. Many of the new users are not as interested or knowledgeable about the instrumentation and are more interested in rapid turnaround. This necessitates a rethinking of some past practices. For example, dedicated (single use) beamlines are becoming the norm for protein crystallography stations. The NSLS is considering expanding this practice where possible and forming consortia to build and operate such stations. The UEC is monitoring the process closely and encourages users to give us feedback on the these trends.

Quarterly town meetings sponsored by the UEC are held in the NSLS seminar room and the UEC meets on the same day to provide opportunities for users to voice their concerns. While most of the attendees at these meetings are frequent users, local beamline contacts and NSLS staff, everyone is welcome. With the NSLS and BNL staff in attendance, there are opportunities for immediate discussion and feedback. The UEC also separately meets with NSLS and BNL staff and management to discuss relevant issues. A main issue of concern this year has involved changes in lab wide computer security and its effect on users. Fortunately, Carl Eyler (eyler@bnl.gov), the new head of cyber security was at a recent Town Meeting to directly address the critical issues and to provide a contact point for problems. Another major change for the users this year was the permanent shift to the higher ring energy of 2.8 GeV with reduced emittance. This was a development studied and endorsed by the UEC some time ago and it is good to see it implemented. Other issues studied and endorsed by the UEC include upgrades to the ring and beamlines suggested by the NSLS and termed Phase III. Although the DOE has failed so far to endorse these upgrades as a package, some capital funds have been pledged and the UEC will be looking to advise the NSLS management as to what, within Phase III, can be accomplished and where it sits on the user's radar screens. The UEC has also reached out to other user groups in the DOE synchrotron community, meeting with the heads of the user organizations from ALS, APS, and SSRL and organizing a lobbying trip to Washington, DC to support the DOE budget. These activities were considered successful and will be continued.

Lastly, the UEC runs the NSLS annual meeting. A report of the 2000 meeting appears elsewhere in this volume and hopefully you are reading this while you are attending the 2001 meeting! The annual meetings continue to be very successful with 250-300 attendees and 7 workshops this year. They reflect a vigorous user community and the vibrant science going on at the NSLS. As of the publication of this activity report, I am passing on the baton to Simon Bare, the new UEC Chair. I have enjoyed greatly the job of guiding the UEC and I know Simon will do a great job for you this year. But remember, he can't do a good job unless you tell him what's going on and what your concerns are. So call or e-mail him (srbare@uop.com), come to the Town meetings and the User's meeting, or run for election to the UEC or as Special Interest Group representative. It will be a good use of your time and will help improve the NSLS for us all.

Users' Executive Committee (including Special Interest Group Representatives): Lisa Miller, Larry Carr, Michael Dudley, Shane Stadler, Mary Anne Corwin, Barbara Illman, Mark Chance, Tony Lanzirotti, Mark Lucas, Leemor Joshua-Tor, Michael Vaughan, Simon Bare, Chris Jacobsen, and Kenneth Evans-Lutterodt.

Users' Meeting Planning Committee: Mark Chance, Linda Feierabend, Chris Johnson, Mary Anne Corwin, Simon Bare, Nancye Wright and Peter Stephens.

NSLS 2000 Annual Users' Meeting and Workshops

Mark Chance

Users' Executive Committee Chair, Albert Einstein College of Medicine

The National Synchrotron Light Source (NSLS) annual meeting was recently held at Brookhaven National Laboratory (BNL) on May 22-24, 2000. Over 260 registered attendees participated in 5 workshops, a poster session, a vendor exhibit, lectures, a reception and a banquet.

The festivities began Monday the 22nd with workshops on Environmental and Geological Sciences and X-ray Absorption Fine Structure Studies of Dilute Systems. As the 19 exhibitors set up their displays in the lobby of Berkner Hall, students and Post-Docs set up posters in the adjoining rooms, hoping to win cash prizes for 1st, 2nd or 3rd place in the poster competition. At 5:30, a welcoming reception and the poster session began, with live music and an open bar. Old friends got reacquainted and the author even met someone he knew in high school and hadn't seen in 25 years, who was in the synchrotron game!

On Tuesday, the main meeting began with an update of activities at BNL by Dr. John Marburger, BNL Director. Dr. Marburger painted an upbeat picture with the opening of the new high energy physics Relativistic Heavy Ion Collider facility (RHIC) and the thousand or so new users that will be regularly coming to BNL. Although the closing of the High Flux Beam Reactor was admitted as a serious blow, the importance of the Light Source within the lab framework as a "multi-facility" lab was emphasized.

Dr. Marburger then introduced the new BNL Associate Laboratory Director for Basic Energy Sciences, Richard Osgood, Jr., a Professor of Physics from Columbia. Dr. Osgood oversees both the NSLS and the Chemistry Department and started his new position only in the last month with the retirement of Denis McWhan. Dr. McWhan served as the ALD for Basic Energy Sciences from 1995 to 2000 and as the NSLS Chairman from 1990 to 1995. Although, Dr. Osgood's expertise is primarily in lasers, his group has used the synchrotron and he was responsive to the concerns of a large user facility as well as to the need for new directions.

After Dr. Osgood, the audience was taken on a virtual ride of protein crystallography beamline X12C worthy of Disneyland. Using dual projection, a live 'telepresence' and remote operation, Dr. Robert Sweet of the Biology Department showed us all how it can be done from the low bandwidth comfort of your living room. The demonstrated remote capability that Dr. Sweet and colleagues have developed with support from the National Center for Research Resources of the National Institutes of Health (NIH) immediately generated discussion of what other beamlines these techniques could be transferred to and how this could be expanded.

Subsequent to this first scientific talk, Michael Hart, Chairman of the National Synchrotron Light Source, gave an overview of another very good year, while yours truly, outlined how the User's Executive Committee (UEC) is working hard to make the case for increased synchrotron funding in Washington, D.C. This included a joint visit by the UEC leadership of all 4 DOE synchrotrons to lobby members of Congress on April 11, 2000. In UEC elections, Simon Bare, Leemor Joshua-Tor, and Michael Vaughan were elected to 2-year terms with Simon Bare assuming the position of vice chair of the UEC. Along with Mark Chance, Simon will take over these lobbying activities in future years.

Excellent scientific highlights outlining the potential uses of femtosecond x-rays (Janos Hajdu, Uppsala University) and the potential for generating them (Igor Pogorelsky, BNL) rounded out the morning's activities. Dr. Hajdu described imaging biological materials with femtosecond coherent pulses that will be an important scientific application of new fourth generation sources. NSLS's Igor Pogorelsky described the promising potential for generating these rays using the BNL Accelerator Test Facility (ATF). In a proof of principle experiment, Pogorelsky's team had produced the highest photon yield ever demonstrated via laser Thomson scattering on relativistic electron beams. As he reported, in the next experiment, they expect to produce x-ray pulses a thousand times more intense and shorter than the conventional synchrotron sources. This will enable a new spectrum of multi-disciplinary, ultrafast x-ray studies and applications.

After outdoor lunch under the tent, Arthur Bienenstock, Associate Director for Science of the Office of Science and Technology Policy, gave us a peek into the inner workings of Presidential science advising. He emphasized the real concern in the Administration with respect to the funding lag for the non-biological sciences that has occurred over the last 25 years. The audience was reminded that only their direct efforts can effect change.

The day concluded with an NSLS Science Advisory Committee (SAC) User Forum, chaired by Sol Gruner of Cornell University, where users posed questions about future directions for the NSLS. The SAC advises the NSLS Chair on the present status and future directions of NSLS programs. With the user community and NSLS staff, they are exploring more ways to support the expanding number of NSLS users. Ideas included new beam lines for environmental and biological sciences, new accelerator or free electron laser sources for BNL, as well as maintaining the momentum of the NSLS as the most used synchrotron source in the world. A lively discussion outlined these exciting directions and strong comments from the SAC about the health and strength of the facility. After the forum, the attendees retired to Giorgio's Restaurant for a banquet. Some attendees tried the valet parking with mixed success.

The meeting concluded Wednesday with additional workshops on Very Bright Infrared Sources and Applications, New Approaches to Solving Protein Crystal Structures, and Chemical Applications of Synchrotron Radiation (see accompanying articles in this issue of SRN). Overall, the meeting was a great success, both well attended and very informative.

Environmental and Geological Sciences Workshop

Barbara Illman[1] and Sue Wirick[2]

[1]U.S. Department of Agriculture, Forestry Service, [2]SUNY at Stony Brook

For years scientists have been using synchrotron light to analyze soils, minerals and organic matter. The ability to analyze very small amounts of wet samples for elemental concentrations as low as parts per billion, to map those elements, and obtain oxidation states has great advantages over more conventional methods of analysis. The discussions in this workshop focused on work using XAFS [X-ray Absorption Fine Structure], XANES [X-ray Absorption Near Edge Structure], XRF, [X-ray Fluorescence], and imaging to examine several broad topics; the mobilization and containment of radionuclides in the natural environment; the interaction of soils with heavy metals; the microbial oxidation of transition metals in soils and in remediation of lignocellulose in wood; and the degradation of plant matter.

Cleveland Dodge along with A. J. Francis and Jeff Gillow [Brookhaven National Laboratory] have been investigating DOE's Waste Isolation Plant Project [WIPP] where they are studying the dissolution, precipitation, and mobilization of transuranics in soils by microbes. Dodge uses XAFS to determine the molecular structure of transuranic organometallics formed by microbes in various radionuclide wastes. One can use some species of bacteria to accumulate up to 15% of U in solution. Their ongoing research is to understand the microbial transformations of actinides so a realistic evaluation can be made of long-term impacts on the storage of radioactive wastes.

Martine Duff presented research conducted by herself and Doug Hunter [Savannah River Ecology Laboratory], D. L. Bish and D. J. Vaniman [Los Alamos National Laboratory] and A. Manceau [U. of Grenoble, France] on the study of geochemical processes that control Pu adsorption to mineral phases in heterogeneous geological materials [tuffs]. Using XANES and XRF they looked at tuffs from Yucca Mountain range in Nevada. This area is being evaluated as a potential subsurface site for the storage of nuclear wastes. Duff et al have found that some trace minerals, specifically MnO, can sorb contaminants such as Pu, changing the Pu's solubility and mobility by altering Pu's oxidation state. These highly reactive fracture fill minerals are important in the sorption and transport of contaminants in the natural environment, even at the low overall concentration of MnO at <1%.

Richard Reeder [Geosciences Dept., SUNYSB] has been studying the uptake of uranium by calcium carbonate minerals. Calcite, in particular, is a ubiquitous phase, found in sedimentary, metamorphic and igneous environments, as well as in soils like those at the Hanford Site, south-central Washington. Carbonate minerals are highly reactive and may be important for sequestration of actinide species. Using XAFS, Reeder has found that uranyl carbonate species coprecipitated with the metastable mineral aragonite show coordination similar to that in the aqueous solution from which it grows. In contrast the coordination of the uranyl species in calcite is different, suggesting a more complex mechanism of incorporation. Reeder also used micro-XAS to show that incorporation of uranium at the calcite surface is highly heterogeneous, being controlled by the distribution of different surface sites.

Darrell Schulze described the research he is conducting at X26A in collaboration with Chris Guest [Agronomy Dept., Purdue U.], Ian Thompson and Don Huber [Botany & Plant Pathology Dept., Purdue U.]. This team is using XRF and micro-XANES spectroscopy to study the role of Mn redox chemistry on plant fungal diseases, specifically take-all in wheat and blast in rice. The Mn redox chemistry in soils plays an important role in plant uptake of Mn and movement of trace elements associated with Mn-oxide minerals, thereby affecting plant health. XANES spectroscopy and extraction data suggest that the main form of Mn(II) in reduced soil is an amorphous Mn-carbonate or a Mn-phosphate. The team designed and built sample cells that allow spatially resolved XANES and XRF spectra from soils at the interface between bulk soil and the surface of live plant roots. Mn(II) remains homogeneously distributed in bulk soil. In water saturated soil the fungus *Gaeumannomyces graminis* accumulates and oxidizes Mn(II) to insoluble Mn(IV) thus making it possible for this wheat disease fungus to induce Mn deficiency in plants prior to invasion.

George Cody [Geophysical Laboratory, Carnegie Inst., Washington DC], using XANES and image mapping of carbon functional groups, discussed the degradation of carbon in wood by microbial degradation and geological processes. The two common groups of fungi degrading modern trees preferentially attack different components of wood. White rot fungi degrade lignin while brown rot fungi primarily degrade cellulose. Cody has found that white rot fungi are very efficient at consuming almost all of the lignin present in the sample. In collaboration with Andrew Knoll and Kevin Boyce [Harvard U., MA] Cody has also looked at ancient woods, some as old as 450 million years, and found the carbon in these woods to be in an organic phase, in the forms of both carbohydrate and lignin.

William Bleam [Department of Soil Sciences, U. of Wisconsin-Madison] presented his work on the effect of sulfur on the behavior of mercury in the environment: Hg(II) and methylmercury cation. Though much about the chemistry in the environment remains a mystery, monitoring at the landscape scale identifies organic matter and sulfur as two important players influencing mercury dynamics. Understanding the role of organic matter and sulfur, however, presents significant challenges because it demands an uncommon level of discrimination. First, we must distinguish and quantify the sulfur species in the environment to identify those that may react with mercury. Second, we require methods that reveal the structure and composition of mercury or methylmercury complexed by organic matter. Synchrotron-based x-ray absorption spectroscopy, as it turns out, satisfies both of these demands. XANES supplies quantitative information on the forms of sulfur while XAFS reveals both the structure and composition of the binding sites. While humic thiol ligands are important, the behavior of the methylmercury suggests a more complicated story awaits us.

Barbara Illman (USDA Forest Service, Forest Products Lab, U. of WI) uses synchrotron methods for environmental, forest ecosystem, forest products and tree disease research. She presented studies on chromium redox chemistry in wood and wood waste treated with the preservative, chromated copper arsenate (CCA). Several strategies are being developed for management of treated waste. Micro-XANES and XRF were used to determine chromium location and redox states in wood during bioremediation and degradation. The studies provide needed information about the fate of Cr(III) and the more toxic Cr(VI) in wood and about the effect of bioremediation by metal-tolerant fungi.

XAFS Studies of Dilute Systems Workshop

Kumi Pandya
North Carolina State University

A workshop on "XAFS Studies of Dilute Systems" was held at the National Synchrotron Light Source (NSLS) on May 22, 2000. Over 40 participants attended this workshop. Seven scientific talks addressing recent developments in X-ray detectors and XAFS applications to dilute systems were presented. Topics included biological, chemical, material and environmental sciences.

Professor Grant Bunker, Illinois Institute of Technology, presented the first talk reviewing various detectors used for the fluorescence mode XAFS measurements. He discussed problems encountered at the third generation synchrotron radiation sources where very high photon fluxes saturate the solid state detectors. Professor Bunker described design principles of two devices that selectively reject the scattered background photons before they reach the detector - thereby preventing the detector from getting saturated. The first device, an array of synthetic multilayer, is being used at the BioCAT beamline of the Advanced Photon Source (APS). The second device uses extremely bent, thin silicon crystals in Laue geometry, developed for medical imaging at the NSLS.

Professor Doug Pease, University of Connecticut, continued on the topic of detector developments. He discussed the capabilities and limitations of a Log Spiral of Revolution (LSR) detector covered with highly oriented pyrolytic graphite for detecting the fluorescence XAFS of an element in the presence of competing fluorescence from other elements present. His data included fluorescence XAFS for several binary alloys. For a $Cr_{.80}Mn_{.20}$ sample the LSR can be tuned to accept the fluorescence either from Cr only or from both Cr and Mn.

Dr. Ken Kemner of Argonne National Laboratory presented a talk about the application of sub-micron X-ray beams to study dilute systems in environmental biogeochemistry. He emphasized that the microenvironment at an actively metabolizing cell surface can be significantly different from the bulk environment. Dr. Kemner presented the results of his team's XAS and microimaging studies at microscopic level to determine the spatial distribution and chemical speciation of Cr associated with *Pseudomonas fluorescens* (a common soil microbe).

Dr. Joe Woicik, National Institute of Standards and Technology (NIST), presented a talk about EXAFS of thin semiconductor alloys and oxides. For a thin Gd_2O_3 film epitaxially grown on the GaAs(001) substrate, the Gd-O bond length shows a 2.7% increase relative to the bulk Gd_2O_3. This increase in the bond length corresponds to a lattice strain of e[110] = +4.8% perpendicular to the Gd_2O_3/GaAs(001) interface.

Professor Bruce Gates, University of California at Davis reviewed the pros and cons of various conventional methods used for synthesizing supported metal catalysts and the spectroscopic methods used for characterizing them. Professor Gates and coworkers have synthesized catalysts from organometallic precursors to obtain highly uniform metal clusters. The in-situ EXAFS studies of several systems were presented including MgO-supported Re subcarbonyls, Ir_4/Al_2O_3, Ir_6/Al_2O_3, Rh_6/NaY and Pt/Al_2O_3

Professor Dean Hesterberg, North Carolina State University, reported on EXAFS and Micro-XANES investigations of metal sulfide oxidation in soil. It is very difficult to characterize the metals in soil because the geochemical systems are spatially heterogeneous. They successfully combined the bulk EXAFS with Micro-XRF and Micro-XANES techniques to determine the spatial distribution of copper and zinc sulfides in contaminated soils. The results show that the metal sulfidation-oxidation rates depend on soil chemical processes occurring on a micro-scale.

Dr. William O'Grady, Naval Research Laboratory, presented the last workshop talk, discussing the XANES investigations of chloride interactions with passivated aluminum films. These films were formed by anodically polarizing the Al samples at a series of potentials below the pitting potentials. The results indicate that the Cl^- migrates from the solution/Al oxide interface into the passive Al oxide film, prior to pit initiation, as a function of the applied potential gradient. Once a critical anodic potential is reached, the oxide undergoes dissolution. The ingress of Cl^- into the oxide is a precursor to localized oxide failure and pitting corrosion.

Very Bright Infrared Sources and Applications Workshop

Gwyn Williams
Thomas Jefferson National Laboratory

The NSLS has hosted several infrared (IR) workshops in the past, including one at each of the past 3 user meetings. This time the scope was extended to include all accelerator produced IR sources and experimental programs. The meeting has grown each year and this year the program barely fitted into one day. The workshop was attended by over 50 participants representing every IR facility in the US as well as strong representation from several countries in Europe.

Paul Dumas (LURE, Orsay, France) gave the opening talk which set an excellent tone for the meeting. LURE has the largest suite of facilities, including a linac based IR-Free Electron Laser (FEL), a storage-ring based near-UV FEL, as well as IR synchrotron facilities. Highlights from all the IR programs were presented including surface dynamics via sum frequency generation and high resolution microscopy. One slide showed both FEL and IR beams (naturally synchronized) at a sample in the microscope. Strong collaborations with Brookhaven National Laboratory (BNL) have yielded many papers and some of this work was also presented. Lisa Miller (BNL) then gave a talk summarizing the evolution of infrared applications to biological materials and presenting specific highlights from the rapidly expanding number of programs at the National Synchrotron Light Source (NSLS). Lisa included data from the new Spectra-Tech Continuum microscope recently installed at Beamline U10B. A contrast from synchrotron work was then presented by Todd Smith from the Stanford FEL at which an impressive suite of experimental programs has developed over many years, a recent highlight being some new near-field microscopy. We then moved to Europe for a summary by Mark Surman of the IR synchrotron programs at Daresbury, including some very impressive state-of-the-art spectra with noise down to 0.01%, and a suite of surface science and microspectroscopy. Lindsay Keller (MVA Inc.) then presented a detailed study of interplanetary dust particles using IR microspectroscopy in conjunction with other techniques using other NSLS beamlines, showing the advantages of a synchrotron facility.

After an excellent lunch and discussions in the conference room, Fred Dylla (Jefferson Lab, VA) introduced the newest user facility at the high average power FEL and showed examples of recent user data. This FEL uses superconducting technology and electron beam re-circulation to generate light of average power in the kilowatt regime. In addition, the infrared photons scatter from the electrons to produce X-rays with pulse lengths of a few hundred femtoseconds.

The meeting continued with a switch back to synchrotrons with Mike Martin showing the wide range of experiments that are taking place at the Advanced Light Source, Berkeley. He also spoke about beamline developments and future prospects at this exciting facility. One of the highlights of the meeting was the talk by James Allen, Director of the Quantum Institute at the University of California at Santa Barbara. The FEL at this center has generated an impressive number of basic research programs and these were eloquently detailed with a focus on the fundamental physics that has motivated them. Medical and other programs developed at the Vanderbilt FEL and now continuing at the Duke FEL were then presented by Glenn Edwards. Glenn also discussed the full suite of facilities at Duke, which include an FEL and a storage ring with an FEL, thus opening up many possibilities for pump-probe spectroscopy. These were further discussed later in the program by Shane Hutson (Duke University) who outlined plans to build an IR beamline on the storage ring, automatically synchronized with the FEL as at LURE.

David Ernst, Director of the Vanderbilt FEL facility, then gave a presentation with detailed transparencies showing the medical programs and in particular the applications of IR-FEL light to tissue ablation with minimal colateral damage. Recently the work has extended to human subjects and Vanderbilt has plans for a considerable expansion of its programs in the future.

The day ended with a series of shorter talks, the first one being from Dave Schiering (Spectra-Tech) on the latest instrumentation developments in IR microspectroscopy. These have culminated in a fine new instrument, and Dave discussed technical and optical issues. Alex Goncharov then presented the exciting physics that occurs at high pressures, and summarized recent work by the Geophysical Laboratory of the Carnegie Institution of Washington at a new beamline (U2A) which they have built at the NSLS. Finally, continuing with the theme of new science with new facilities at the NSLS, Laszlo Mihaly presented new data taken with a Sciencetech SPS-200 Martin-Puplett Polarizing Interferometer. This forms the first part of a new facility that includes a magnet for Electron Spin Resonance (ESR) studies.

Following a lively dinner at a local Chinese restaurant, the workshop continued with 2 days of hands-on microscopy. Several participants took advantage of free access to all the latest instrumentation from the NSLS, the Albert-Einstein College of Medicine, and the Carnegie Institution. Particular thanks are due to Lisa Miller, Larry Carr, Ned Marinkovic and Zhenxian Liu for giving their time and expertise to make this so useful.

As organizer, I would like to express my thanks to Simon Bare and to all the people behind the scenes at the NSLS, particularly Linda Feierabend, who made this event run so smoothly and, for me, so effortlessly.

New Approaches to Solving Protein Crystal Structures Workshop

Zbigniew Dauter
Science Applications International Corp.

During the workshop six invited speakers presented their results. They encompassed both the new theoretical ideas and novel practical approaches to phasing diffraction data from crystals of macromolecules.

In line with the context of ever more synchrotron beam lines available worldwide for protein crystallography, the first speaker, Wladek Minor (Univ. of Virginia, Charlottesville) presented a software system for remote control of a diffraction experiment through the Internet. It is based on the popular data processing package HKL2000, and allows the experimenter located in the home laboratory to proceed with data collection at a distant synchrotron beam line with simultaneous processing of diffraction images at his laboratory. This approach requires only the shipment of frozen crystals to the synchrotron site and is particularly relevant for structural genomics, where rapid turnover is crucial.

The first step in the process of solving novel structures by multi- or single-wavelength anomalous diffraction data is the location of anomalous scatterers. One of the most powerful direct methods used for this purpose is SnB. Dave Smith (Hauptman-Woodward Institute, Buffalo) described the newest version of SnB and explained how to get the most out of it. Indeed, in large structures it is capable of finding more than a hundred selenium sites. Only a few years ago crystal structures with more than a hundred atoms in total were recognized as an ambitious project.

The most popular way of introducing anomalous scatterers to protein crystals for subsequent MAD phasing is through exchange of methionines by selenomethionines. K. Rajashankar (Rockefeller University and NSLS) presented the new approach, based on derivatization by short soaking of crystals in a cryoprotecting solution containing bromide or iodide ions. Halides adopt ordered sites at the protein surface and can be utilized for phasing by MAD (bromides) or SIRAS (iodides). This approach is simple and requires a minimal amount of time and effort, and therefore can be relevant for high throughput projects. It has been successfully applied when classic heavy-atom derivatization proved negative and incorporation of selenomethionines impossible.

Zygmunt Derewenda ((Univ. of Virginia, Charlottesville) discussed the simplification of the classic approach to MAD phasing. He has presented results of successful structure solutions against single wavelength data, utilizing only the Bijvoet differences as a source of phasing. The outstanding experimental electron density map of 56 kDa structure of human acyl protein thioesterase was obtained from the anomalous signal of 22 bromide ions cryosoaked into the crystals with the use of single wavelength data collected at the bromine absorption edge. The results of SeMet MAD phasing against data from various numbers of wavelengths suggests that it may be more advantageous to limit the number of wavelengths utilized but improve data quality by increasing the multiplicity of measurements.

Ribosomes belong to the most complicated and largest macromolecular structures ever solved by X-ray crystallography. Nenad Ban (Yale University) described the ingenious methods used to solve the structure of the 50 S ribosome particles and extend the resolution of the analysis beyond 3 Å, which is unprecedented for such a large complex of proteins and nucleic acids. The wealth of information from the ribosome model at the atomic level is overwhelming, as is the beauty of its structure.

Tom Terwilliger (Los Alamos Natl. Laboratory) presented the novel maximum likelihood application to the density modification process. Density modification is used to improve the quality of experimental electron density maps, often obtained from weak anomalous and/or isomorphous signals of heavy atoms. Several examples of a dramatic improvement of map quality were presented and the new program, soon to be released, will certainly gain very wide popularity.

The workshop was a great success in providing an overview of recent results and significant improvements to the theory and practice of phasing macromolecular structures.

Chemical Applications of Synchrotron Radiation Workshop

Jose A. Rodriguez and Jan A. Hrbek
Brookhaven National Laboratory

The workshop “Chemical Applications of Synchrotron Radiation” was held during the main NSLS 2000 Users’ Meeting on May 24 at BNL. The number of attendees surprised organizers and forced them to scramble for additional chairs to accommodate everybody interested.

Professor T.K. Sham (UWO, London, Canada) started the morning session with a discussion of synchrotron radiation (SR) advantages and by summarizing recent trends and developments in chemical applications. In the second part of his talk he demonstrated the use of site-selective spectroscopy using soft x-ray photons with emphasis on an optical technique, x-ray excited optical luminescence (XEOL) in studies of porous silicon and Si nanowires, and organic light emitting device materials such as Alq_3 (tris-8 hydroxylquinoline aluminum).

Dr. Dave Mullins (ORNL) made a strong case for the use of Soft X-ray Photoelectron Spectroscopy. Examples he presented ranged from studies of dilute components, the evolution of surface intermediates in real time, and the elucidation of vibrational structure in the photoemission of adsorbed molecules. He also discussed potential experimental problems that result from having an intense, tunable source (calibration of excitation energy, diffraction effects, sample charging radiation damage).

Professor Jingguang Chen (U Delaware) demonstrated the critical importance of the NEXAFS technique for the investigation of the unique physical and chemical properties of transition metal carbides (TMC) and nitrides (TMN). These TMC/N often have catalytic advantages over their parent metals in activity, selectivity and resistance to poisoning. In addition, they often show catalytic properties that are characteristic of the expensive Pt-Group metals. Measurements at the C and N K-edges are very useful for the characterization of carbides and nitrides and for studying their behavior under different chemical environments.

Dr. Fritz Hoffmann (SCI-MED) discussed surface chemistry studies performed with infrared radiation from the U4-IR beamline and demonstrated quite unique applications of time-evolved Fourier Transform Infrared Reflection Absorption Spectroscopy (te-FT-IRAS) and IR microscopy to the characterization of intermediates in important catalytic reactions. Specifically, he presented te-FT-IRAS data collected during the adsorption of molecules relevant to the epoxidation of butadiene on Ag and the potassium promoted activation of CO_2. Finally, IR microscopy was applied to coal chemistry by characterizing the spatial distribution of the various chemical components in small coal particles (20-100 µm in diameter).

Dr. Jia Wang (BNL) demonstrated the utility of x-ray scattering techniques in surface electrochemical studies. She talked about the self-assembly of polyoxometalates on metal surfaces and formation of molecular nanostructures having interesting electronic, catalytic and structural properties. Specifically, the silicotungstate anion (STA), $SiW_{12}O_{40}^{4-}$, as a model system, was studied on Ag (100) and Ag (111) by using surface x-ray scattering techniques. From analysis of specular reflectivity, configuration specific adsorption of STA was found on Ag (100) and Ag (111). These data indicate that maximizing the number of Ag-O contacts plays a major role in the STA adsorption.

Prof. Clare Grey (SUNY SB) followed with a presentation under the descriptive title “Combined Diffraction and NMR Studies of Catalysis and Sorption: Hydrofluorocarbon and Ethylene Sorption and Catalysis Reactions over Zeolites, Halo-Zeotypes and Aluminas”. These studies illustrated the utility of time-resolved X-ray diffraction for identifying the active structure of catalysts, adsorption sites in zeolites and phase transitions in the solid state.

Dr. John Larese (BNL) gave a spirited description of the molecular adsorption and surface mediated chemical reaction studies using a combination of materials synthesis, x-ray and neutron and thermodynamic methods. In particular, he illustrated the effectiveness of this multifaceted approach for studying the interaction of sulfur and nitrogen oxides with pure and doped MgO. He discussed how diffraction measurements at X7B and XANES near the sulfur edge using X19A at NSLS were extremely useful for identifying the structure and chemical nature of the adsorbed species.

Structural Genomics at the NSLS: NIH Awards Major Grants to Brookhaven National Laboratory

Mark Chance

Users' Executive Committee Chair, Albert Einstein College of Medicine

The National Institute of General Medical Sciences (NIGMS) has developed a major new initiative to determine the structures of thousands of proteins over the next decade. Work toward this goal will be divided into two phases: a 5-year pilot stage and a subsequent 5-year full-scale production phase. The initial phase began on September 27 with the announcement of funding for Structural Genomics centers.

BNL and NSLS staff and beamlines are closely involved with two of the centers, which were awarded after an international competition. The first, the New York Structural Genomics Research Consortium, includes a partnership between five New York institutions including: the BNL Biology Dept., which runs crystallography beamlines X12B and X12C, Albert Einstein College of Medicine, which runs X9A and X9B, as well as Rockefeller, Mt. Sinai, and Cornell Weill Medical College. The TB Structural Genomics Consortium will also utilize beamline X-8C through connections with LANL.

“These research centers are true pilots,” said Dr. John Norvell, director of the NIGMS Protein Structure Initiative. “Each will include every experimental and computational task of structural genomics and will develop strategies for use in the subsequent large-scale research networks. By the fifth year of the award, we expect each pilot center to reach a production level of 100 to 200 protein structures annually, which is significantly greater than the current rate of protein structure determination.” Users may remember that Dr. Norvell was the keynote speaker at the NSLS Annual meeting in 1999. Based on the generous funding for the structural genomics centers, and the key role to be played by the NSLS, it appears that Dr. Norvell remembers us.

BNL's Science & Technology Awardees Honored in 2000

Diane Greenberg and Karen McNulty

For their development of structural biology beamlines at the National Synchrotron Light Source (NSLS), Lonny Berman, NSLS Department; Malcolm Capel, Biology Department; and Robert Sweet, Biology, shared a Science & Technology Award. Each of the three scientists was honored for contributions to designing instrumentation at NSLS structural biology Beamlines X8C, X12B, X12C, X25 and X26C and were also cited as a resource for the hundreds of users who conduct their research at these beamlines each year.

Top: Malcolm Capel, Marshall Newton. Bottom: Lonny Berman, Laurence Littenberg, Bob Sweet. Not present: David Cox.

Additionally, they were commended for the award because, under Sweet's guidance, they secured a five-year, $8.3 million grant from the National Institutes of Health (NIH) National Center for Research Resources in 1998, matching the U.S. Department of Energy funding for this type of research at the NSLS. The grant has allowed Berman, Capel, Sweet and others to develop new structural biology techniques, equipment, and software to be used at the five beamlines. It also allows for the hiring of new staff and the implementing of new research projects in structural biology. Using x-ray crystallography at the structural biology facilities, scientists can determine the atomic structure of biological molecules, such as proteins, viruses and DNA complexes. From this information, they can learn about how these molecules function, which is important in understanding disease mechanisms, the immune system, and heredity. The data are also used in pharmaceutical development and in the bioengineering of plants. Recent discoveries at the five structural biology beamlines include images of part of the Lyme disease bacterium interacting with a part of the human immune system.

Also, landmark progress in understanding ribosome structure was reported in two papers in Nature. Capel was a collaborator in both ribosome research projects, and Sweet was a collaborator in one of them. Berman first came to BNL as a student research assistant during the summer of 1980, while he was an undergraduate student at Cornell University. In 1987, he joined Brookhaven as an assistant scientist in the NSLS Department, and, two years later, he became an associate scientist. He was promoted to physicist in 1998. Michael Hart, NSLS Chair, commented: "Lonny Berman has been in charge of the X-25 beamline since its inception. It is a world-class beamline, with an extraordinary record of performance. Lonny is also a key member of the construction team for our next NIH-funded NSLS project - building a structural biology station for NIH on Beamline X6." Capel came to Brookhaven in 1982 as a postdoctoral fellow at Yale University, and, in 1985, he was hired as an assistant biophysicist in the Biology Department. In 1987, he became an associate biophysicist, and he was promoted to Biophysicist in 1991. Sweet was an assistant professor at the University of California at Los Angeles (UCLA), and a specialist in molecular biology at UCLA¹¹s Molecular Biology Institute before he joined Brookhaven's Biology Department as a scientist in 1983.

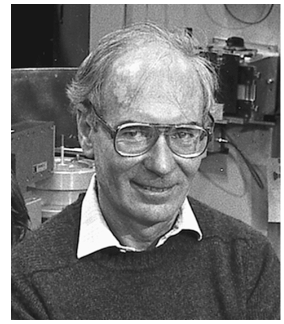
In nominating Sweet and Capel for this award, Carl Anderson, Biology Department Chair, said that Sweet's guidance was crucial, in collaboration with Capel and others, in obtaining the NIH five-year award for the NSLS structural biology facilities. Anderson added that these facilities are currently the most productive in the world. Further, Anderson gave Sweet credit for developing Beamline X12C, which, he said, "has become in many ways a gold standard for beamlines around the world, in both ease of use and quality of data collected."

Anderson further noted that Capel's "initial responsibilities were to complete a small-angle station at X12B, which had been designed and partly constructed by others before him. He substantially redesigned the station, developed software for controlling it, and produced an excellent facility, which is used by a wide variety of researchers."

Senior physicist David Cox was honored for his contributions to materials science and synchrotron powder x-ray diffraction, and for the design and operation of state-of-the-art spectrometers at the National Synchrotron Light Source (NSLS) and the High Flux Beam Reactor (HFBR).

Cox came to Brookhaven in 1963 as an associate physicist. In his career, he has used neutron powder diffraction techniques to determine the ordered arrangements of atomic magnetic moments in countless materials, including one of the earliest oxide superconductors and many high-temperature superconductors. Cox also recognized in the early 1980s that synchrotron x-rays could be a powerful tool for powder diffraction work, and did seminal experiments to demonstrate its superior capabilities in comparison to then standard methods.

He went on to build and operate an extremely productive spectrometer for powder diffraction studies at the NSLS. This work depended greatly on his ability to get funding and forge collaborations with outside researchers, including scientists at Mobil, Dupont, Air Products, the Carnegie Institution as well as universities. Through these efforts, Cox has been a leader in structure-property studies of zeolites (important catalysts in the oil industry), copper-oxide superconductors, giant magnetoresistance compounds, and Bucky-ball compounds. In the midst of his growing career, Cox took time out to design the High Resolution Powder Diffractometer at the HFBR. He was tenured in 1987 and retired this January.



David Cox

"Cox's achievements are recognized worldwide," said Physics Department Chair Michael Murtagh. "His efforts have made BNL a center of excellence in powder crystallography, and have brought in many important outside collaborators. We'll miss his talent, creativity, and dedication."